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16. ABSTRACT

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Mass Spectrometric Quantification of Polychlorinated Biphenyl Congeners Using Multiple Carbon-13 Internal Standards

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Multiple carbon-13 internal standards improve the precision and accuracy of the mass spectrometric analysis of polychlorinatedbiphenyl (PCB) congeners compared to analyses using a single carbon-13 internal standard. The mean percentage error in PCB congener analysis on the $\mu g\ g^{-1}$ level using multiple ion detection and three carbon-13 internal standards was 6.29%. This precision in single laboratory work suggests that the method will be useful for interlaboratory studies monitoring PCB residues and contaminants of public health significance.

INTRODUCTION

Polychlorinated biphenyls (PCBs) are present in the environment as residues from technical mixtures, biological modifications of technical mixtures, and from various (over 40) different chemical reactions.[1] Animal studies have shown that the individual PCB congeners vary in toxicity and distribution. One evidence of the congener-specific nature of the distribution of PCBs is that human blood samples lack the early eluting congeners of commercial PCB formulations.[2] As a result of the differences in the fate of different congeners, accurate quantification of total PCBs and of individual PCB congeners requires a congener-specific method.

A recent review describes the regulatory history and analytical problems of PCBs.[3] Since 209 congeners are possible and many environmental samples contain chlorinated pesticides which interfere with some of those congeners, even capillary column gas chromatography (GC) does not give satisfactory resolution. All 209 PCB congeners have been synthesized and analyzes using capillary GC with electron capture detection have been reported.[4] However, electron capture detection is nonspecific and subject to interferences, and the responses of the congeners vary so widely that an accurate calculation of total PCBs in a sample would require use of a different response factor for each PCB congener present. Gas chromatography/ mass spectrometry (GC/MS) has been shown to offer an additional level of specificity when operated in the electron impact (EI) mode and less variation of response factor is found within congeners of the same chlorine number.[5,6]

The response of PCB congeners in GC/MS analysis using single ion monitoring has been reported previously.[7-10] Techniques for response data collection have included monitoring only the molecular ion of the PCB analyzed and that of the internal standard, the use of multiple ion detection (MID) scans that cover the region of interest and full scans, 20 to 670 a.m.u.[10] Response factors were calculated based on a comparison of one ion of the molecular ion cluster[7,8] or the entire molecular ion cluster to the standard.[9,10] Within a given set of PCB isomers, the mass spectral response factors have varied up to a factor of two.

Validation of the capillary GC/MS analysis of PCBs requires the inclusion of procedures to achieve acceptable precision and accuracy. Based on previous GC/MS work, multiple internal standards[11,12] and carbon-13 labeled counterparts[13,14] were selected to aid analysis. A sample containing a known representative group of PCB congeners (performance evaluation sample) was obtained from the EPA repository for this study.

EXPERIMENTAL

Neat PCB congeners were acquired as analytical standards from the EPA Toxic and Hazardous Materials Repository, Austin, TX. A 1 mg or larger sample of each congener was weighed to ±0.01 mg and diluted with chlorobenzene (Burdick and Jackson Laboratories, Inc., Muskegon, MI) to give three standard solutions of different concentration levels. The EPA Repository also furnished a mixture, '^{13}C-PCB Recovery Surrogates, SS100,'† which contained 99% isotopically pure, >98.4% chemically pure, $^{13}C_{12}$ congeners 212, 213 and 214 in hexane (see Table 1). This solution also contained congener 211, (1',2',3',4',5',6'-$^{13}C_6$)4-chlorobiphenyl diluted to 1.9$\mu g\ ml^{-1}$ in the final standard mixture. On

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† This standard can be obtained free of charge by Office of Toxic Substances Contract Laboratories and Government Laboratories with the permission of John Smith, Office of Toxic Substances, Washington, DC. Commercial standards are available from Cambridge Isotope Laboratories, Inc., Woburn, Ma.

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Table 1. Composition of standard mixtures

Congener number[9,10]	Compound	Levels (μg ml^{-1})		
77	3,3',4,4'-Tetrachlorobiphenyl	14.5	7.3	1.5
153	2,2',4,4',5,5'-Hexachlorobiphenyl	46.0	23.0	4.6
138	2,2',3,4,4'5'-Hexachlorobiphenyl	10.6	5.3	1.1
167	2,3',4,4',5,5'-Hexachlorobiphenyl	11.2	5.6	1.1
183	2,2',3,4,4',5',6-Heptachlorobiphenyl	12.3	6.2	1.2
185	2,2',3'4,5,5',6-Heptachlorobiphenyl	11.7	5.9	1.2
170	2,2',3,3',4,4',5-Heptachlorobiphenyl	12.7	6.4	1.3
202	2,2',3,3',5,5',6,6'-Octachlorobiphenyl	45.0	22.5	4.5
204	2,2',3,4,4',5,6,6'-Octachlorobiphenyl	16.7	8.4	1.7
198	2,2',3,3',4,5,5',6-Octachlorobiphenyl	13.3	6.7	1.3
194	2,2',3,3',4,4',5,5'-Octachlorobiphenyl	16.9	8.5	1.7
206	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	19.4	9.7	1.9
209	Decachlorobiphenyl	17.0	8.5	1.7
212	3,3',4,4'-Tetrachlorobiphenyl (Carbon-13 Internal Standard)	5.1	5.1	5.1
213	2,2',3,3',5,5',6,6'-Octachlorobiphenyl (Carbon-13 Internal Standard)	8.1	8.1	8.1
214	Decachlorobiphenyl (Carbon-13 Internal Standard)	10.0	10.0	10.0

spiking 1 ml of each of the three standard solutions with 20 μl of the carbon-13 standard, the standard mixtures shown in Table 1 were obtained.

A performance evaluation sample of known PCB congeners in 1:1 chlorobenzene: 1,2-dichlorobenzene was supplied by the EPA Repository for Toxic and Hazardous Materials, Austin, TX.

A Finnigan 4023 quadrapole mass spectrometer was used for analysis. The instrument was tuned to manufacturer's specifications with FC 43. Final tuning criteria[16] for 50 ng of injected bis(pentafluorophenyl)phenylphosphine was met each day over the five-day acquisition period. The analysis conditions were: Fused silica DB-5 column (J & W Scientific, Rancho Cordova, CA) 1.0 μM film thickness, 0.25 mm × 15 M; helium linear velocity 30 cm s^{-1}; injector temperature 250 °C; and column temperature program 120 °C for 1 min then 8 °C min^{-1} to 325 °C. One μl aliquots were injected in the Finnigan Model 9610 gas chromatograph using a Grob-type injection. The column was interfaced directly to the ion source operating at 70 eV and 270 °C.

In the full scan (FS) mode, the mass range 95–550 was scanned in 1.00 s with 10 centroid samples per peak. In the multiple ion detection (MID) scan, the mass ranges were selected to include the molecular ion clusters of the selected congeners with the actual dwell times shown in Table 2.

Table 2. Multiple ion detection (MID)/Scan

Interval	Mass range	Dwell time (s)
1	287.5 to 294.5	0.046
2	299.5 to 306.5	0.046
3	355.5 to 364.5	0.059
4	385.5 to 401.5	0.105
5	425.5 to 434.5	0.059
6	437.5 to 446.5	0.059
7	459.5 to 468.5	0.059
8	493.5 to 504.5	0.072
9	505.5 to 516.5	0.072

Table 3. Quantification masses for determining PCB congeners

Congener molecular formula	Ion mass
$^{13}C_{12}H_6Cl_4$	304
$C_{12}H_6Cl_4$	292
$C_{12}H_4Cl_6$	360
$C_{12}H_3Cl_7$	394
$13C_{12}H_2Cl_8$	442
$C_{12}H_2Cl_8$	430
$C_{12}HCl_9$	464
$^{13}C_{12}Cl_{10}$	510
$C_{12}Cl_{10}$	498

Multiple internal standards were selected with retention time windows for PCB congeners from 0.8 to 1.2 times the retention time of the internal standard with three internal standards required for this range of PCB congeners. Three internal standards were selected to cover the range of PCB congeners analysed. Each congener was compared with the internal standard of nearest retention time; in all cases the congener retention time was within 0.8 to 1.2 times the retention time of the selected internal standard. The performance evaluation sample was analysed and quantified using Finnigan Autoquan software with both three internal standards and one internal standard versus the standard concentrations. Quantification was done using mean response factors. Response factors were calculated as (congener area/congener amount)/(reference area/reference amount). Triplicate injections of each of the three standard mixtures (Table 1) were made using FS and MID modes. The ion mass quantified for each congener is shown in Table 3.

The concentrations of the PCBs in the performance evaluation sample were within the concentration ranges of the standards and no detectable impurities were observed in the chlorobenzene or the standards.

Regression analysis was performed on a VAX 11/750 using the Statistical Analysis System (SAS, Inc.) data package. Significance tests were performed manually at the 5% significance level using statistical estimates of slope, intercept and standard error provided by SAS.

RESULTS AND DISCUSSION

The analysis of the three standards described in Table 1 resulted in four sets of mean response factors (RFs) and % relative standard deviations (RSDs), depending on scan mode (MID or FS) and three internal standards (1 IS or 3 IS), as shown in Table 4. An important feature of use of three internal standards is that with the carbon-13, carbon-12 pairs it is possible to determine the precision and accuracy of the mean RF values. For example, for the 3 IS, MID scan, mean RF values, the response factor percentage difference was: Tetrachloro-(congener 212 versus 77), 0.70%; octachloro-(213 versus 202), 1.98% and decachlorobiphenyl (214 versus 209), 2.96%. This is a strong indication that no significant saturation or detection problems are present.

Decachlorobiphenyl for the 1 IS, MID scan mode, gave a response factor difference for the carbon-13,

Table 4. Variation of mean response factors of standards, MID or full scan modes, one or three internal standards, single ion quantification

Congener number	Compound	MID 3 IS RF	MID 3 IS RSD (%) N=9	MID 1 IS RF	MID 1 IS RSD (%) N=9	FS 3 IS RF	FS 3 IS RSD (%) N=9	FS 1 IS RF	FS 1 IS RSD (%) N=9
212	3,3',4,4'-Tetrachlorobiphenyl Carbon-13	1.000	0.00	1.000	0.00	1.000	0.00	1.000	0.00
77	3,3',4,4'-Tetrachlorobiphenyl	0.993	3.68	1.007	5.08	1.003	2.06	1.012	4.99
153	2,2',4,4',5,5'-Hexachlorobiphenyl	0.711	3.16	0.719	4.32	0.779	2.56	0.778	2.65
138	2,2',3,4,4',5'-Hexachlorobiphenyl	0.523	7.50	0.523	7.51	0.540	15.90	0.545	15.92
167	2,3',4,4',5,5'-Hexachlorobiphenyl	0.602	6.49	0.595	5.98	0.632	17.03	0.617	21.74
183	2,2',3,4,4',5',6-Heptachlorobiphenyl	0.387	7.74	0.390	8.80	0.356	16.81	0.355	19.29
185	2,2',3,4,5,5',6-Heptachlorobiphenyl	0.818	6.83	0.418	9.93	0.808	20.94	0.385	21.51
170	2,2',3,3',4,4',5-Heptachlorobiphenyl	0.903	2.51	0.474	7.00	0.851	12.96	0.407	14.90
213	2,2',3,3',5,5',6,6'-Octachlorobiphenyl Carbon-13	1.000	0.00	0.518	3.86	1.000	0.00	0.494	1.28
202	2,2',3,3',5,5',6,6'-Octachlorobiphenyl	1.020	3.80	0.529	2.34	1.041	3.94	0.514	3.50
204	2,2',3,4,4',5,6,6'-Octachlorobiphenyl	1.051	2.54	0.538	6.68	1.034	10.65	0.503	10.76
198	2,2',3,3',4,5,5',6-Octachlorobiphenyl	0.750	6.40	0.386	11.01	0.728	15.54	0.350	14.14
194	2,2',3,3',4,4',5,5'-Octachlorobiphenyl	1.183	4.48	0.433	11.29	1.140	16.91	0.386	18.44
206	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	0.841	11.01	0.318	21.05	0.827	25.53	0.265	29.77
214	Decachlorobiphenyl Carbon-13	1.000	0.00	0.349	7.95	1.000	0.00	0.308	5.98
209	Decachlorobiphenyl	1.030	3.18	0.388	9.90	0.948	9.40	0.297	13.88
	Mean RSD (%)		5.33		8.36		13.10		14.73

carbon-12 pair of 10.6%. This large variation in response factor for decachlorobiphenyl is reasonable as it has the lowest RF and was at the lowest concentration of all the carbon-13 standards.

Inspection of the RSD values shown in Table 4 shows the degree to which three internal standards improves the precision for the response factors obtained by MID and FS. Comparing the variability (RSDs) between MID 3 IS and 1 IS RSD values shows that 11 of 13 RSD values are lower for the 3 IS RFs and the average improvement is 3.03%. With FS data, 11 of the 13 RSD values are again lower for the 3 IS RFs with an average improvement of 1.63%. Precision is improved with the 3 IS, MID acquisition mode RF giving an excellent mean RSD of 5.33%.

In this work the full scan spectra on the carbon-12 congeners, at the lowest concentration level, generally did not have the expected natural abundance carbon-13 isotope peaks. Some isotope peaks were actually absent. The lowest concentration was at the limit of instrumental sensitivity for full scan. All other FS (and MID) spectra (or partial spectra) did show reasonable carbon-13 isotope peaks. Regression analysis was done to compare the MID data with the FS data as plotted in Figs 1 and 2.

The 3 IS data were linear, fitting the model $RF_{FS} = \beta_0 + \beta_1 RF_{MID} + \varepsilon$. Constant β_0 is the RF_{FS} intercept. β_1 is the slope and ε is the residual error. For the 13 data points the estimator value $\hat{\beta}_0$ was 0.0394 with a standard error of 0.0412, and the estimator value $\hat{\beta}_1$ was 0.9411 with a standard error of 0.0478. The correlation coefficient was $r = 0.9861$. β_0 is not significantly different from zero ($t = 0.9563$) and β_1 is not significantly different from one ($t = -1.2322$). For the 1 IS data using

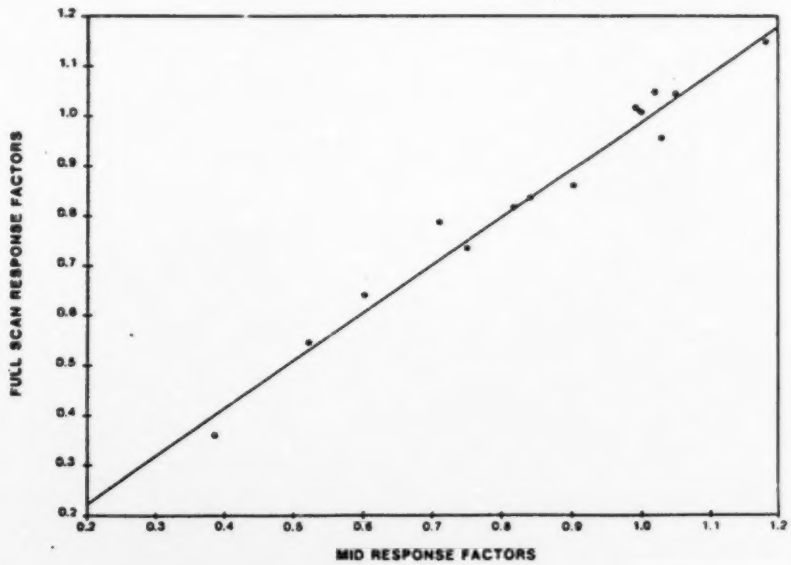

Figure 1. Mean response factors–MID versus full scan with three internal standards.

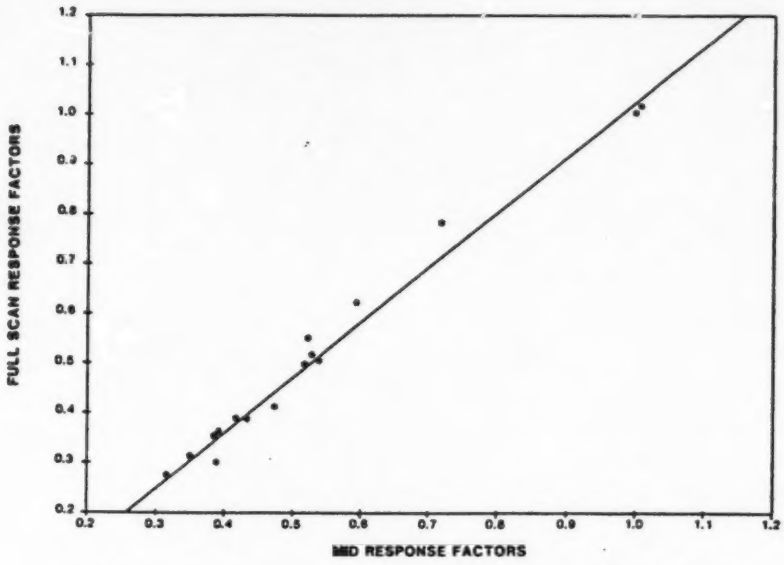

Figure 2. Mean response factors–MID versus full scan with one internal standard.

the model $RF_{FS} = \beta_0 + \beta_1 RF_{MID} + \varepsilon$, 15 data points, $\hat{\beta}_0 = -0.0959$ with a standard error of 0.0250, $\hat{\beta}_1 = 141$ with a standard error of 0.0470, $r = 0.9892$. β_0 is significantly different from zero ($t = -3.83$) and β_1 is significantly different from unity ($t = 3.00$). The estimates β_1, β_0, and r and the standard errors of β_1 and β_0 were obtained directly from SAS output. The significance tests were performed manually using the t-statistics

$$t = \frac{\hat{\beta}_0 - 0}{s_0} \quad \text{and} \quad t = \frac{\hat{\beta}_1 - 1}{s_1}$$

where s_0 = the standard error of $\hat{\beta}_0$, and s_1 = the standard error of $\hat{\beta}_1$. Thus, the RFs from FS and MID scan modes agree; the 3 IS approach gave better agreement than 1 IS. With full scan data, 1 IS, octachlorobiphenyl isomers, there is a relatively large range of RSD values. The precision was generally worse for those isomers with lower than average response factors.

Table 5 shows the results of the analysis of the performance evaluation sample analysed using the response factors of the standards and the Finnigan Autoquan software. The mean percentage error using three internal standards and MID scan is 6.29%. The percentage error observed in the MID scan using one internal standard was greater and was about 20% for those congeners (194 and 206) with the greatest difference in retention time from the internal standard (congener 212).

In all cases the single ion used in quantification was the base peak of the spectrum. Most spectra were available in the EPA/NIH or CIS Mass Spectral Data Bases, and matched well with the experimental spectra. The three spectra not available from these data bases are shown in Fig. 3.

Horwitz[17,18] suggests that analyses, at the $\mu g\ g^{-1}$ level with a practical analytical method for monitoring residues and contaminants of public health significance should have a reproducibility of <16% for interlaboratory studies. Corresponding single laboratory variation would typically be 8 to 11%.

The practical method for mass spectrometric analysis of PCB congeners on the $\mu g\ ml^{-1}$ level revealed by this

Table 5. Concentration of performance evaluation sample quantified using mean response factors

Congener number		Prepared concentration ($\mu g\ ml^{-1}$)	MID 3 IS Level found ($\mu g\ ml^{-1}$)	MID 3 IS Error (%)	MID 1 IS Level found ($\mu g\ ml^{-1}$)	MID 1 IS Error (%)	FS 3 IS Level found ($\mu g\ ml^{-1}$)	FS 3 IS Error (%)	FS 1 IS Level found ($\mu g\ ml^{-1}$)	FS 1 IS Error (%)
153	2,2',4,4',5,5'-Hexachlorobiphenyl	5.20	4.99	4.04	4.93	5.19	4.43	14.81	4.44	14.62
138	2,2',3,4,4',5'-Hexachlorobiphenyl	3.96	4.18	5.56	4.18	5.56	4.16	5.05	4.12	4.04
167	2,3',4,4',5,5'-Hexachlorobiphenyl	7.10	7.84	10.42	7.93	11.69	8.48	19.44	8.69	22.39
183	2,2',3,4,4',5',6-Heptachlorobiphenyl	5.71	6.48	13.49	6.44	12.78	7.03	23.12	7.07	23.82
185	2,2',3,4,5,5',6-Heptachlorobiphenyl	7.96	8.32	4.52	7.86	1.26	9.17	15.20	9.76	22.61
170	2,2',3,3',4,4',5-Heptachlorobiphenyl	4.06	3.76	7.39	3.46	14.78	4.02	0.99	4.26	4.93
204	2,2',3,4,4',5,6,6'-Octachlorobiphenyl	7.90	7.35	6.96	6.93	12.28	8.58	8.61	8.99	13.80
198	2,2',3,3',4,5,5',6-Octachlorobiphenyl	6.97	6.50	6.74	6.09	12.63	6.99	0.29	7.42	6.46
194	2,2',3,3',4,4',5,5'-Octachlorobiphenyl	4.96	5.03	1.41	4.00	19.35	4.61	7.06	5.36	8.06
206	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	9.97	10.21	2.41	7.84	21.36	9.45	5.22	11.58	16.15
	Mean % error			6.29		11.69		9.98		13.69

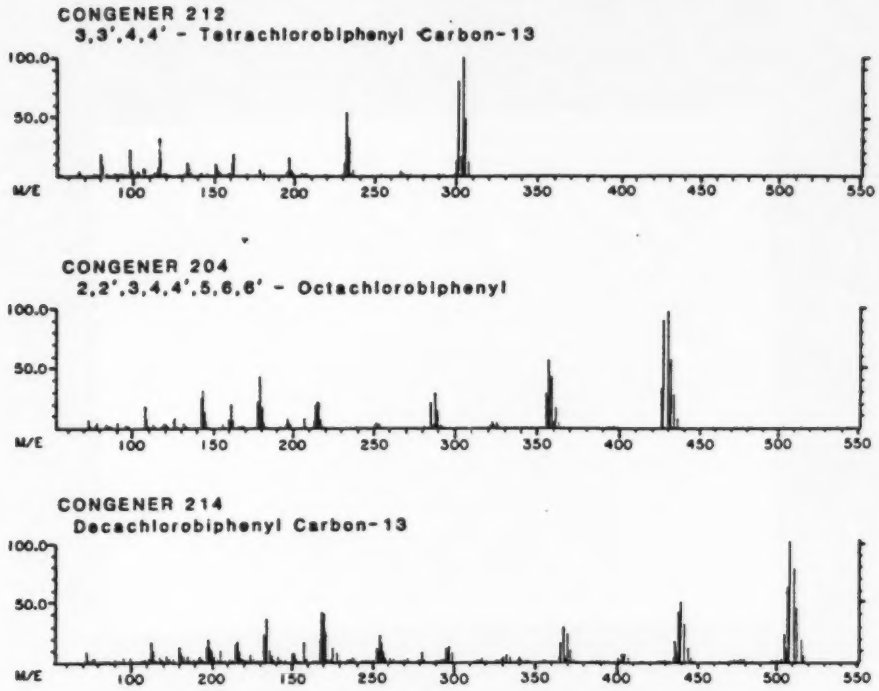

Figure 3. Full scan spectra of selected PCB congeners.

study requires MID scanning and multiple carbon-13 internal standards.

The response variation inherent to the mass spectrometer or to discrimination by the capillary injection system[19] is corrected for, over the range of analytes by the use of multiple carbon-13 internal standards.

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